MORPHOLOGY OF DEFORMED ZONE OF MICRO- AND NANOINDENTATION ON CRYSTALS WITH DIFFERENT TYPE OF BOND

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Abstract

The comparison of microindentation (MI) and nanoindentation (NI) methods has been made. Differences between nanohardness ($H_n$) and microhardness($H_m$) values were found. The deformation zone around these two types of indentations, the dislocation structure and the thin structure of indentation relief have their specific peculiarities, which were attributed to the scale effect and different deformation extent of material under MI and NI.

1. Introduction

Last years, under conditions of rapid development of nanotechnologies, the nanoindentation method has obtained a wide application due to the possibility to study the elastic-plastic properties of size-limited materials, such as thin film, fibers, heterostructure, nanocrystals, etc. In addition, this method opens new opportunities for the investigation of physical nature of mechanisms, responsible for strength and plasticity of small volume of materials. At the same time, during last decades extensive scientific material was accumulated in the field of microindentation: the stress state, dislocation structure and surface morphology of deformed zone around the indentation, there were proposed the models of material plastic flow, etc. [1]. In this connection it will be useful to compare these two methods for the purpose to reveal common and distinctive peculiarities, that will further their more correct use.

2. Experimental technique

A wide range of crystals KCl, LiF, MgO, CaF$_2$, GaP, Si with different type of bond, from typically ionic to typically covalent, was chosen for investigation. This choice was stipulated for comparison and generalization of obtained results. Indentations were made on (001) plane for KCl, LiF, MgO, GaP crystals and (111) planes for CaF$_2$ and Si, using the Nano Indenter II (MTS Systems, USA) with diamond trihedral Berkovich pyramid as indenter and loads $P_{\text{max}} = (0.01 \div 0.12) N$, and Microindenter PMT-3 with diamond tetrahedral Vickers pyramid as indenter and loads $P = (0.01 \div 2.0) N$. Nanohardness was determined using the standard method described in [2], microhardness was determined from the known formulae $H = 1.854 \frac{P}{d^2}$, where $d$ – is the indentation diagonal. The thin structure of surface relief of indentations was investigated using Atomic-force microscope (AFM) (trademark Nanostation) and the dislocation zones around the indentations were revealed using etch-pit technique.
3. Results and discussions

The calculation of microhardness ($H_m$) and nanohardness ($H_n$) values showed that nanohardness is higher than microhardness for all investigated crystals (Table 1). Besides, within the limits of one method the hardness is not constant as well and increases with the load decrease for both MI and NI (Fig. 1, 2a). Such hardness – load dependence $H$ ($P$) is consistent with the earlier studies on a wide range of materials for MI [3] and recent work on KCl for NI [4]. Figure 1 shows, that harder materials exhibit more pronounced hardness increase with load decrease. MgO crystal exhibits a small drop of hardness for $P<0.2N$. One can see, that with load decrease the values of microhardness approach those of nanohardness. The hardness – indenter penetration dependence $H(h)$ (Fig.2a) was obtained by special method, described in [5], using standard load – penetration curve (Fig.2b).

Table 1. Comparison of microhardness ($H_m$) and nanohardness ($H_n$) values.

<table>
<thead>
<tr>
<th>Crystal</th>
<th>$H_m$, GPa ($P=1.0N$)</th>
<th>$H_n$, GPa ($P=0.01N$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>KCl</td>
<td>0.10</td>
<td>0.16</td>
</tr>
<tr>
<td>LiF</td>
<td>1.23</td>
<td>1.46</td>
</tr>
<tr>
<td>MgO</td>
<td>8.43</td>
<td>10.77</td>
</tr>
<tr>
<td>CaF$_2$</td>
<td>1.53</td>
<td>1.99</td>
</tr>
<tr>
<td>GaP</td>
<td>9.0</td>
<td>11.78</td>
</tr>
<tr>
<td>Si</td>
<td>6.12</td>
<td>13.15</td>
</tr>
</tbody>
</table>

The effect of hardness increase with load decrease may be connected with the so-called *scale effect*. The volume of deformed zone for typical loads at NI is many times smaller than at MI. The significant increase of deformation localization can lead, in our opinion, to the reduction of presence in this zone of already existent dislocations in real crystal and the necessity of new dislocation generation, that need much greater stresses. In these conditions, the deformation of super-small volumes of crystal may be approximated to the deformation of ideal crystal. This assumption is confirmed by the results, obtained in work [6], where the dislocation loop nucleation was registered using nanoindentation test and the stresses of this nucleation are very close to theoretical critical stresses of dislocation generation.

The examination of deformed and dislocation zone around the indentation showed that, as a whole, the shape of indenter does not affect the form of dislocation rosettes (Fig.3). This fact means that the situation of active slip planes has here a major influence: {110}$<110>$ slip system for KCl, LiF, MgO and {100}$<110>$ slip system for CaF$_2$. The dislocation zone around the indentations on Si GaP is situated very close to the indentation and can’t be revealed around the indentations made at room temperature, using etch-pit technique.

At the comparison of the dislocation rosettes of microindentations and nanoindentations for the same loads, a small discrepancy was found in the lengths of screw arms ($l_s$) on MgO crystals, namely, they are longer for NI (Fig.3 a, b). The lengths of edge arms ($l_e$) have very
close values. The ratio $l_s/l_e$ is 0.85 – for MI and 1.0 – for NI. Possibly, this discrepancy results from the different type of used indenter.

One more difference was observed in the deformed zone around MI and NI on CaF$_2$ crystal (Fig. 3 c, d). The indenters made at the same load – 0.09N by MI method are absolutely plastic and by NI exhibit pronounced crack formation. This event appears to be correlated to different extent of deformation created by Vickers and Bercovich indenter.

![Diagram](image)

Fig. 2. LiF, NI; a – nanohardness - indenter penetration dependence, b – load - penetration dependence.
Fig. 3. The dislocation rosettes around the indentations on (001) plane of MgO (a, b) and (111) plane of CaF$_2$ (c, d); a, c - MI, Vickers indenter; b, d - NI, Bercovich indenter; P, N: a - 1.0; b, c, d - 0.09.

The calculation of deformation extent produced by each of indenters was made from the geometry of ideal indenter, using the formulae [7]:

$$\varepsilon = \frac{(A_s - A_{pr})}{A_{pr}} \times 100\%,$$

where $A_s$ is the area of indentation surface, and $A_{pr}$ is the project area of indentation. The results showed that for Bercovich indenter the deformation extent $\varepsilon_B = 10.4\%$ and for Vickers indenter $\varepsilon_V = 7.7\%$. Besides, the different sharpness of indenter tip induces additional influence on the deformation extent of material. Namely, the tip radius $r_v \approx 500 \text{ nm}$ for Vickers indenter means that for penetration depth $h < 60 \text{ nm}$ in fact only the spherical part of indenter works, which deformation extent drop gradually from 7.7\% to 0 with $h$ diminution. For Bercovich indenter with tip radius $r_B \approx 40 \text{ nm}$ the deformation extent $\varepsilon_B = 10.4\%$ is achieved already at $h = 4 \text{ nm}$. Therefore at the initial stage of indenter penetration Bercovich indenter produces several times higher extent of deformation than Vickers indenter. All this leads to the formation of stronger dislocation agglomerations, higher stresses which cause the crack formation.

The found discrepancies in thin structure of surface relief for MI and NI, in our opinion, are also connected with the different deformation extent of material under Vickers and Bercovich indenters. For all investigated material the surface of indentations was found to be not smooth but covered by so-called facets. The NI exhibits more pronounced formation of facets (Fig. 4). Analogical formation of facets was revealed for the first time in [8] by compression of thin plate of NaCl and was attributed to the rotation deformation of material when usual deformation by slip is inconvenient. Hence, the higher deformation extent of material around the nanoindentations can lead to greater contribution of rotation deformation to general deformation process of indentation.
4. Conclusions

It was found that besides common regularities of deformation by concentrated load – stress state, active slip planes for concrete crystal, the deformed zones around the micro- and nanoindentations have their specific peculiarities. The principal reasons causing the observed differences between MI and NI are:

- **type and sharpness of indenters**, resulting in higher deformation extent for NI, which leads to the crack formation and more pronounced formation of facets;
- **scale effect**, causing the discrepancy in values of micro- and nanohardness.

Acknowledgements

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References